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Hurwitz

[11] **3,870,590**
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[54] **NON-FLAMMABLE POLYESTER TEXTILE
ARTICLES AND METHODS FOR MAKING
THEM**

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260/80.77**

[51] Int. Cl. **D03d 1/00**

[58] Field of Search **161/88, 151, 92, 156, 170,
161/231, 403, 150, 175; 117/161, 138.8;
260/DIG. 24, 80.77, 80.6; 57/140 BY**

[56]

References Cited

UNITED STATES PATENTS

2,783,215	2/1957	Robitschek	161/403
3,516,903	6/1970	Jones et al.	161/175
3,658,579	4/1972	Ottinger et al.	161/170
3,829,532	8/1974	Meloy et al.	260/DIG. 24
3,833,535	9/1974	Wambach	260/DIG. 24

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[57]

ABSTRACT

Non-flammable textile articles of polyester fibers are prepared by blending flammable polyester fibers with non-flammable fibers and then applying a sizing or binder of a non-flammable polymer which sizing may contain a flame retardant synergist.

16 Claims, No Drawings

NON-FLAMMABLE POLYESTER TEXTILE ARTICLES AND METHODS FOR MAKING THEM

Polyester compositions including fibers formed therefrom, such as those obtained from the products in U.S. Pat. Nos. 2,465,319 and 3,047,539 are readily flammable and have the disadvantages that they tend to drip while burning or being subject to flames and it is difficult to prevent the dripping. Common flame retardant additives, such as phosphorous compounds and antimony compounds do not appear to be very effective when used with polyesters. Polyesters are also subject to serious degradation in the presence of a number of conventionally used flame retardants with a loss in physical properties.

It has been known that flameproof fibers and filaments can be made from various polymeric film-forming materials. Generally, fibers made from such materials are quite expensive and unsuitable for many uses. Such fibers have been blended with filaments or fibers of a flammable nature in an attempt to obtain textile products having non-flammable properties. The fibrous products obtained from such a mixture of polyester fibers still have deficiencies making them unsuitable for many uses if the proportion of non-flammable fibrous content is high enough to make the products self-extinguishing. For example, non-flammable blends of a fiber formed of a halogen containing polymer with a polyester type of fiber generally contain from about 50 to about 65 percent by weight of the former type of fiber (unless the blend also contains a large amount of inorganic non-combustible materials) and textile products made therefrom are deficient in certain properties. In woven and non-woven textiles, the hand is frequently deficient. Also, the use of conventional polymeric binders for sizing a woven fabric or for bonding the fibers of a non-woven web tends to increase the flammability of the textile products. In the production of bulky fibrous masses especially fiberfill which is used in apparel, for example, linings for suits, padded garments, such as bedjackets and smoking jackets, insulated garments, such as sports jackets, bedding, such as quilts, for upholstery padding and cushion fillers, including automotive protective padding, such as "headliners" in the ceiling or on the dashboards of steering wheels, the relatively large proportion of halogen containing polymer imparts a severe loss of resiliency and resulting tendency to pack down in use.

In accordance with the present invention from about 5 to 35 percent of a non-flammable halogen containing fiber is blended with from about 95 to about 65 percent of a polyester fiber to afford products which when subjected to extreme heat will not melt or burn. The products are used to prepare woven fabric or non-woven webs, batts or mat of fibers employed in apparel as a textile fabric or as a liner or padding material, as a stuffing material for upholstering purposes and the like. In making a woven textile, a polymeric sizing material is employed and comprises from about 5 to 20 percent by weight based on the weight of fibers. This sizing is a polymer composition which even when normally dried or cured or both on the textile material to form part of the finished textile product, does not support combustion. Similarly, in making a bulky non-woven material for use as an upholstery stuffing, filler for cushions or a non-woven textile suitable for use as a fabric, such as a curtain, drapery and the like or as a

lamina of a multi-ply textile for use in garments, for example, shirt collars, cuffs or linings for mens' suits from about 5 to about 50 percent by weight of a polymer binder composition based on fiber weight which when dried and cured does not support combustion, is used as a binder for bonding the fibers together.

One preferred embodiment of the present invention is concerned with fibrous products comprising a major proportion of polyester fibers obtained from terephthalates such as those prepared from terephthalic acid and ethylene glycol, dimethyl terephthalate and ethylene glycol, dimethyl terephthalate and 1,4-cyclohexanedimethanol and the like. Tradenames of various products prepared from terephthalate include Dacron, Terylene, Kodel, Diolen, Enkalene, Fortrel, Tergal, Terital, Terlenka, Trevira and the like which are of the thermoplastic character. These fibers are blended with from about 5 to about 35 percent by weight based on the total weight of the fiber blend, of a halogen containing polymer, such as homopolymers and copolymers of vinyl chloride, vinylidene chloride, vinyl fluoride, vinylidene bromide, tetrafluoroethylene and the like. Preferably, the fiber blend comprises a mixture of from about 5 to about 35 percent by weight of halogen containing thermoplastic fibers and from about 65 to about 95 percent by weight of thermoplastic polyester fibers. Non-woven fabrics are made from these blends by bonding the fibers distributed in random array with a polymer composition which contains a substantial amount of halogen, preferably chlorine, in the prepolymer.

It has been found that by employing non-flammable fibers, in the range of from about 5 to about 50 percent and preferably in the range of from about 5 to about 35 percent, which contain from about 10 to about 60 percent by weight of a halogen such as chlorine and bromine and having a shrinkage temperature of at least 250°F. and preferably above 300°F., in the blend with the polyester fibers, together with a sizing or binding polymer composition containing from about 60 to about 85 percent by weight of vinylidene chloride prevents dripping of melted fiber on exposure of the fiber mass to an open flame. Vinyl chloride, vinyl bromide, vinylidene chloride or vinylidene bromide homopolymers and copolymers as well as vinyl chloride containing modacrylic fibers and vinylidene chloride containing modacrylic fibers such as those disclosed in U.S. Pat. No. 3,516,903 may be used in the fiber blend.

The chlorine containing polymer fiber which is blended with the polyester must be of the type that will not shrivel into a knotty mass nor shrink more than about 15 percent lengthwise (measured on the length of the individual fiber) when subjected to the drying and curing temperatures employed on a sized woven fabric or a fiber-bonded non-woven product. These products are used as textile fabrics or as stuffing in upholstery. Preferred polymers include syndiotactic polyvinyl chlorides such as one sold under the trademark Leavil; vinyl chloride polymer fibers which heat-set above 300°F., for example, a copolymer containing about 50 percent vinyl chloride and about 50 percent vinyl alcohol sold under the trademark Cordelan; modacrylic fibers formed from copolymers containing from about 35 to about 85 percent by weight of acrylonitrile, 15 to 65 percent of vinyl chloride or vinylidene chloride and up to about 10 percent of other comonomers, such as vinyl acetate, 4-vinylpyridine, methyl acrylate,

ethyl acrylate and the like. A preferred modacrylic fiber is Verel which contains about 50 percent acrylonitrile and about 50 percent vinyl chloride. U.S. Pat. No. 3,516,903 discloses other modacrylic fibers which may be used and said patent is hereby incorporated by reference.

While adequate non-flammability may be obtained by blending a polyester and halogen containing fiber in the proportions specified above, it is often useful to add one or more flame retardants. By adding such retardants the ratio between the polyester fibers and the halogen containing fibers may be increased without loss of non-flammability so that additional variations in properties such as wash durability, durability to dry cleaning, resiliency and desired color may be attained. The coating or bonding copolymers employed in this invention are themselves flame retardants. When an additional flame retardant is employed, such as one or more of those described below, it has been found that there is a synergistic effect in flame retardancy. This synergistic effect is not with only some of the flame retardants mentioned below, but with all of them.

The more important of the flame retardant synergists contain halogen such as bromine or chlorine, antimony, phosphorous or nitrogen. These various synergists and the oxides of various metals including the oxides of antimony, arsenic, aluminum, boron and zinc may be used. Antimony oxide is especially useful for this purpose. The flame retardant additive may also be a halogenated organic compound; a boron containing compound such as zinc borate and the like; a mixture of a halogen organic compound and one of the metal oxides, or a phosphorous compound such as zinc phosphate; a mixture of a halogenated compound with a phosphorous compound or compounds containing phosphorous nitrogen bonds or a mixture of two or more of the foregoing.

The amount of flame retardant will naturally vary with the nature of the fiber blend and with the efficiency of the additive. The amount of additive may be up to about 10 parts by weight per hundred parts of fiber. A preferred amount will be in the range of from about 3 to about 7 parts by weight of additive per 100 parts of fiber.

Among the useful halogen containing compounds that can be used as flame retardants are those described in U.S. Pat. No. 3,671,487, column 4, line 6, to column 5, line 68 inclusive.

Additional non-flammability in the properties of the products can be obtained by introducing flame retardant plasticizers, such as organic phosphate esters, phosphonate esters and the like including tris-(2,3-dibromopropyl)phosphate, tributyl phosphate, triphenyl phosphate, tricresyl phosphate, tris(chloroethyl)phosphate and the like. The plasticizers may be employed up to about 10 percent by weight, based on the weight of blended fibers. Examples of phosphonates which can be employed appear in U.S. Ser. No. 139,949, filed May 3, 1971, which is hereby incorporated by reference.

The fibrous articles of the present invention do not require appreciable amounts of inorganic fillers to ensure that the article is self-extinguishing or that dripping of polymer melt does not occur when the articles are subjected to extreme heat.

The fibers may be blended by equipment commonly used for this purpose, such as by an air-opener by

which small batches of the several different fiber types are introduced in the proportions desired and blown into a chamber by airjets. Other devices that can be employed to form a lap or web of the blended fibers include pickers, cards, garnetts, and Rando-Webber machines. The fibers produced by such equipment are referred to as "randomly distributed" even though there may be some orientation, especially in the carded web. The length of the fibers or filaments may be from about $\frac{3}{4}$ to about 4 inches. In apparels, the preferred length is from about 1- $\frac{1}{2}$ to 2 inches. In some instances, such as in making stuffing materials for upholstering purposes, the length of fiber may be from about 2 to about 10 inches, even up to indefinite lengths (i.e., continuous filaments). Preferably, the length is in the range of from about 1- $\frac{1}{2}$ to about 2 inches plus or minus $\frac{1}{2}$ inch.

From the above described equipment, a web or fleece, mat or batt may be taken and then converted into yarn on a doubler or twister in conventional fashion which yarn can then be woven into a fabric. Alternatively, the product obtained from a card, garnett or "Rando-Webber" may be used in making a non-woven web or lamina, which may be assembled with itself or other laminae, for example, other woven or non-woven materials. These composite laminar fabrics are useful in making curtains, draperies and upholstery stuffing.

Depending on the particular nature of final product desired, the denier of fibers used in the blends are in the range of from about 0.5 to about 20. For use in making upholstery stuffing a denier in the range of from about 5 to about 18 is preferred. Smaller deniers in the range of from about 2 to 5 are preferred for making bonded fiberfill or non-wovens for use in apparel fabrics, curtains, draperies and the like. For special uses, it is often desirable to use a blend of fibers of relatively small denier such as in the range of from 2 to 5, with larger denier fibers, such as those in the range of from 8 to 18.

The coating composition that is applied for bonding fibers in the product comprises a copolymer of vinyl chloride, vinylidene chloride or a mixture thereof with halogen-free comonomers. The proportion of chlorine containing monomers in the copolymer is in the range of from about 60 to 90 percent by weight. The copolymer also contains from about $\frac{1}{2}$ to 5 percent by weight based on the total weight of copolymer, of a reactive comonomer material, including N-methylolacrylamide, N-methylolmethacrylamide, N-methylol-4-pentenoguanamine and the like. The copolymers may also contain up to about 5 percent by weight of other comonomers which have a reactive hydrogen but are not self-reactive, including monoethylenically unsaturated acids, such as acrylic acid, methacrylic acid, itaconic acid and the like, amides of such acids, such as acrylamide, methacrylamide and the like, hydroxy containing esters of such acids, such as β -hydroxyethyl acrylate and β -hydroxyethyl methacrylate, β -hydroxypropyl methacrylate, β -hydroxypropyl acrylate, α -hydroxypropyl acrylate, α -hydroxypropyl methacrylate and the like, aminoalkyl esters of such acids, such as N-dimethylaminoethyl acrylate, N-dimethylaminoethyl methacrylate, and mixtures of two or more of the reactive monomers.

The copolymer may also contain from about 9.5 to about 40 percent by weight of relatively non-reactive monomers, such as alkyl acrylates or methacrylates in which the alkyl has from about 1 to about 18 carbon

atoms and is preferably lower alkyl of from 1 to 8 carbon atoms including methyl, ethyl, n-butyl, hexyl, 2-ethylhexyl, lauryl, stearyl and the like with ethyl acrylate preferred, also other monomers such as vinyl acetate, styrene, acrylonitrile or a mixture of such monomers may be employed.

These copolymers may be applied as a solution or a non-aqueous dispersion in a volatile organic solvent, such as xylene, toluene, benzene, ethylene chloride, acetone, dioxane, or mixtures thereof, at a polymer concentration of from about 1 to about 30 percent by weight. Preferably, the copolymers are applied as aqueous dispersions of water insoluble polymers obtained by emulsion copolymerization of the monomers having a solids concentration in the range of from about 20 to 65 percent as initially prepared.

Application of the coating composition to the fibrous product for sizing or for bonding fibers may be made by spraying the solution or dispersion onto the fibrous product or by immersing the fibrous product into the solution or dispersion at concentrations having a sufficient wet pick-up ratio to provide the desired proportion of polymer on the fibrous product. The product is dried at a temperature in the range of from about 140° to about 200°F. and then cured by heating at a temperature in the range of from about 250° to about 310°F. for from about 1 to about 5 minutes. When thicker products are desired, application by spraying may involve spraying one side of the product, drying it, and then spraying and drying the other side, followed by curing at a temperature and for a time in the range mentioned above.

In general, the relative proportions of the polyester fiber, the halogen containing polymer fiber and the binder copolymer specified above provide a wide range of bonded fibrous products that are flame resistant, flame retardant, or self-extinguishing. However, it is difficult to specify the overall ranges or proportions that will produce a bonded fibrous product that is sufficiently flame-resistant for all purposes in every instance. It is to be understood that the selection of the relative proportions of the polyester and fibers of halogen containing polymer may require (1) the binder polymer to be employed in the lower part of the broad range mentioned above or (2) that the binder polymer contain a proportion of halogen containing monomer that is in the upper part of the range specified. Many factors affect the flame retardance in the fibrous blend, such as size (denier), presence of colorant or delustrant, nature of crimpness, porosity as well as the particular chemical composition of the two fibers. The composition of and relative proportions between the fibers blended and the binder polymer should be correlated to provide the flame resistant characteristics desired in the final bonded product. This normally requires only a simple experiment to check the combination selected.

TEST METHOD

The bonded web sample of about 1/2 to 3/4 inch thickness and having the dimensions of 10 inches by 12 inches is suspended vertically in air having normal room temperature and humidity, for example, 25°C. and 20 to 90 percent relative humidity. A propane gas flame having a length of about 1-1/2 inches is directed into the surface of the hanging web. The web is held in the flame for 5 seconds. This may be done in a protec-

tive enclosure, such as a laboratory hood. The extent of charring, melting, dripping and the time of continued burning after removal of the flame is recorded.

Durability of the sample to washing and dry cleaning is tested by comparing (1) the results obtained on sample swatches of the bonded fibrous articles before any washing or dry-cleaning and (2) the results obtained after subjecting additional swatches of the same bonded article to one or more washing or to one or more dry-cleaning operations or to both washing and dry-cleaning.

For washing, a sample of the bonded web is introduced into the washing machine in individually bagged, 80 sq. bleached cotton sheeting with enough webs or enough terry cloth toweling ballast to provide an 8 pound load in an automatic home washer. The total wash cycle is about 34 minutes including the rinses and spin drying and the solution used in the wash cycle is 15 gallons of water containing one-half cup of the commercially available detergent sold under the trademark "Tide" at 140°F. The total wash cycle is 15 minutes of washing, 5 minutes of spray-spin rinse, a deep rinse at 104°F. for about 2-1/2 minutes and about 3-1/2 minutes dry-spin. The laundering is followed by tumble-drying in a home dryer at the hot setting (about 120° to 140°F.). The drying usually takes about 20 minutes.

For dry-cleaning, the sample is placed in a bag. The bag with its contents is placed into a laboratory model of a dry-cleaning machine containing one gallon of perchloroethylene. The bagged sample is tumbled for about 30 minutes. The bag is removed and the sample web is dried in a tumble dryer at 120°-140°F.

The following examples are illustrative of the invention, the parts and percentages given are by weight and the temperature is Fahrenheit unless otherwise stated.

EXAMPLE 1

A fiberfill blend (garnet) is prepared containing thoroughly distributed in random array, a mixture of 4 parts of 5-denier polyethylene terephthalates (Dacron 88) ranging from about 2 to 2-1/2 inches long and 1 part of 5-denier fibers of vinyl chloride polymer (Leavil) ranging from about 1-1/2 to about 2 inches in length. A 3/4 inch thick, 10 x 12 inch sample of this fiber blend (weighing 13.0 grams) is sprayed on one side with a 23 percent solids dispersion of an emulsion copolymer of 85 percent vinylidene chloride, 9.5 percent methyl acrylate, 3.5 percent ethyl acrylate and 2 percent of an approximately equimolar mixture of acrylamide and N-methylolacrylamide. The sample is dried in a forced-air oven at 240° to 260°F. for about 2 minutes. The other side is similarly sprayed with the same polymer dispersion, dried as before (about 2 minutes at 240° to 260°F.), and then cured for 2 minutes at 300°F. The cured sample weighs 15.1 grams so the amount of the vinylidene chloride binder copolymer therein is approximately 16.1 percent, based on the fiber weight before bonding. When flame-tested in the manner described above with 3/4 inch of the flame extending into the sample, there is no drip or melt even during exposure to the flame. Char length is about 2 inches and burning ceases on removal of the flame.

EXAMPLE 2

By following substantially the procedure of Example 1 and by changing the ratio between the polyester fibers and the polyvinyl chloride fibers from 4:1 to 2:1

and by substituting for the copolymer of Example 1 an emulsion copolymer of 66 percent vinylidene chloride, 30 percent ethyl acrylate, and 4 percent acrylamide and N-methylolacrylamide in approximately equimolar ratio, there is obtained a non-flammable fabric. The add-on of the binder, determined after drying and curing is 16 percent. On flame testing, no melt or drip-burning occurs, the maximum char length is 3-3/4 inches and on removal of the flame, the burning either ceases immediately or within 7 seconds of the removal of the flame.

EXAMPLE 3

By following substantially the procedure described in Example 1 and by substituting for the fiberfill blend described therein a 1:1 blend of polyalkylene terephthalate fibers (Dacron 88) and 5-denier, 2 inches long methacrylic fibers of a copolymer of 50 percent vinylidene chloride and 50 percent acrylonitrile (Verel) there is obtained a fiber blend with a 27.5 percent add-on after spraying the binder of Example 1 which product when exposed to flame in the manner described above neither melts nor drips.

EXAMPLE 4

By employing the blended fiber web prepared in the manner as described in Example 3 and by employing as the bonding copolymer the copolymer described in Example 2, there is obtained a flame resistant fiberfill blend having a 30 percent add on which product when flame tested in the manner described above neither ignites nor melts or drips.

The following table illustrates other blends at various ratios which have been treated with the binder disclosed in Examples 1 or 2 and which also may have been further treated with colloidal antimony oxide (SbO_3) at the 3.0 or 6.3 percent level. The results clearly indicate the non-flammability of the polyester blends both at the stage of original preparation and also after washing and dry-cleaning.

Example No.	Blend	Ratio	Binder of Example	SbO_3	Original Sample	RESULTS	
						After 50 Washes	After Drycleaning
5	*A/B	4/1	1	—	**No 1	***SE-2 seconds	—
6	A/B	10/1	1	6.3%	No 1	SE-1 second	—
7	A/B	5/1	1	3.0%	No 1	No 1	—
8	A/B	1/1	2	—	No 1	No 1	No 1
9	A/C	10/1	1	6.3%	No 1	No 1	—
10	A/C	10/1	1	3.0%	No 1	No 1	—
11	A/C	1/1	1	—	No 1	No 1	No 1
12	A/C	4/1	1	6.3	SE-3	SE-5	—
13	A/D	4/1	1	3.0	SE-1	No 1	—
14	A/D	1/1	2	—	No 1	SE 1, 34	No 1
15	A/D	3/1	2	6.3	No 1	No 1	—

*A - Dacron; B - Leavil; C - Cordelan; D - Verel

**No 1 - no burning on removal of flame after 5 second exposure

***SE - self-extinguishing.

What is claimed is:

1. A non-flammable polyester textile comprised of
 - A. 65 to 95 percent by weight of polyester fibers and
 - B. 5 to 35 percent by weight of fibers of a halogen containing polymer selected from the group consisting of polymers of vinyl chloride, vinylidene chloride, vinyl fluoride, vinylidene bromide and tetrafluoroethylene, coated or bonded to each other with from about 5 to 50 percent by weight,

- based on the weight of fibers, of a copolymer of
 - a. from about 60 to 90 percent by weight of one or more chlorine containing monomers selected from the group consisting of vinyl chloride, vinylidene chloride or a mixture thereof;
 - b. from about 1/2 to about 5 percent by weight of N-methylolacrylamide, N-methylolmethacrylamide, N-methylol-4-pentenoguanamine or mixtures thereof;
 - c. up to 5 percent by weight of other comonomers containing a reactive hydrogen;
 - d. from about 9.5 to about 40 percent by weight of an alkyl acrylate, alkyl methacrylate, vinyl acetate, styrene, acrylonitrile or mixtures thereof; and
- up to about 10 parts by weight of a flame retardant synergist per hundred parts of fiber.

2. A non-woven product according to claim 1 comprised of

- A. 65 to 95 percent by weight of polyester fibers and
- B. 5 to 35 percent by weight of fibers of a chlorine containing polymer selected from the group consisting of polymers of vinyl chloride, vinylidene chloride, vinyl fluoride, vinylidene bromide and tetrafluoroethylene bonded by about 5 to 50 percent by weight, based on the weight of fibers, of a copolymer of

- a. from about 60 to 90 percent by weight of one or more chlorine containing monomers selected from the group consisting of vinyl chloride, vinylidene chloride or a mixture thereof;
- b. from about 1/2 to about 5 percent by weight of N-methylolacrylamide, N-methylolmethacrylamide, N-methylol-4-pentenoguanamine or mixtures thereof;
- c. up to 5 percent by weight of a monoethylenically unsaturated acid, its amide, hydroxyalkyl ester or aminoalkyl ester;
- d. from about 9.5 to about 40 percent by weight of a lower alkyl acrylate, lower alkyl methacrylate, vinyl acetate, styrene, acrylonitrile or mixtures thereof; and

- up to 10 parts by weight of a flame retardant synergist per hundred parts of fiber.

3. A woven product according to claim 1 comprised of

- A. 65 to 95 percent by weight of polyester fibers and
- B. 5 to 35 percent by weight of fibers of a halogen containing polymer selected from the group consisting of polymers of vinyl chloride, vinylidene chloride, vinyl fluoride, vinylidene bromide and

- tetrafluoroethylene, coated or bonded to each other with from about 5 to 20 percent by weight, based on the weight of fibers, of a copolymer of
- from about 60 to 90 percent by weight of one or more chlorine containing monomers selected from the group consisting of vinyl chloride, vinylidene chloride or a mixture thereof;
 - from about $\frac{1}{2}$ to about 5 percent by weight of N-methylolacrylamide, N-methylolmethacrylamide, N-methylol-4-pentenoguanamine or mixtures thereof;
 - up to 5 percent by weight of other comonomers containing a reactive hydrogen;
 - from about 9.5 to about 40 percent by weight of an alkyl acrylate, alkyl methacrylate, vinyl acetate, styrene, acrylonitrile or mixtures thereof; and
- up to about 10 parts by weight of a flame retardant synergist per hundred parts of fiber.
4. The product of claim 2 comprised of
- 65 to 95 percent by weight of a polyester fiber and
 - 5 to 35 percent by weight of fibers of syndiotactic polyvinyl chloride bonded to each other by 5 to 50 percent by weight, based on the weight of fibers, of a copolymer of
- from about 60 to 90 percent by weight of vinylidene chloride;
 - from about $\frac{1}{2}$ to about 2.5 percent by weight of N-methylolacrylamide, N-methylolmethacrylamide, N-methylol-4-pentenoguanamine or mixtures thereof;
 - up to about 2.5 percent by weight of acrylamide, methacrylamide, acrylic acid, methacrylic acid, itaconic acid or mixtures thereof;
 - from about 9.5 to about 40 percent by weight of lower alkyl acrylate, lower alkyl methacrylate, vinyl acetate, styrene, acrylonitrile or mixtures thereof; and
- up to 10 parts by weight of a flame retardant synergist per hundred parts of fiber.
5. The product of claim 3 comprised of
- 65 to 95 percent by weight of polyester fibers and
 - 5 to 35 percent by weight of fibers of a chlorine containing polymer selected from the group consisting of polymers of vinyl chloride, vinylidene chloride, vinyl fluoride, vinylidene bromide and tetrafluoroethylene, bonded to each other by 5 to 20 percent by weight of a copolymer of
- from about 60 to 90 percent by weight of vinylidene chloride;
 - from about $\frac{1}{2}$ to about 2.5 percent by weight of N-methylolacrylamide, N-methylolmethacrylamide, N-methylol-4-pentenoguanamine or mixtures thereof;
 - up to about 2.5 percent by weight of acrylamide, methacrylamide, acrylic acid, methacrylic acid, itaconic acid or mixtures thereof;
 - from about 9.5 to about 40 percent by weight of lower alkyl acrylate, lower alkyl methacrylate, vinyl acetate, styrene or acrylonitrile; and
- up to 10 parts by weight of a flame retardant synergist per hundred parts of fiber.
6. The product of claim 4 wherein the polyester fiber is a terephthalate.
7. The product of claim 5 wherein the polyester fiber is a terephthalate.

8. The product of claim 4 wherein the polyester fiber is derived from terephthalic acid and ethylene glycol, dimethyl terephthalate and ethylene glycol or dimethyl terephthalate and 1,4-cyclohexanedimethanol.
9. The product of claim 5 wherein the polyester fiber is derived from terephthalic acid and ethylene glycol, dimethyl terephthalate and ethylene glycol or dimethyl terephthalate and 1,4-cyclohexanedimethanol.
10. The product of claim 6 wherein the flame retardant synergist contains halogen, antimony, phosphorous, nitrogen or an oxide of antimony, arsenic, aluminum, boron or zinc.
11. The product of claim 8 wherein the flame retardant synergist contains halogen, antimony, phosphorous, nitrogen or an oxide of antimony, arsenic, aluminum, boron or zinc.
12. The method for preparing a flame retardant fiberfill which comprises blending
- 65-95 percent by weight of polyester fibers and
 - 5-35 percent by weight of fibers of a halogen containing polymer selected from the group consisting of polymers of vinyl chloride, vinylidene chloride, vinyl fluoride, vinylidene bromide and tetrafluoroethylene and then treating the blend with from 5-50 percent by weight, based on the weight of the blend, of a copolymer containing
- from about 60 to 90 percent by weight of one or more chlorine containing monomers selected from the group consisting of vinyl chloride, vinylidene chloride or a mixture thereof;
 - from about $\frac{1}{2}$ to about 5 percent by weight of N-methylolacrylamide, N-methylolmethacrylamide, N-methylol-4-pentenoguanamine or mixtures thereof;
 - up to 5 percent by weight of other comonomers containing a reactive hydrogen;
 - from about 9.5 to about 40 percent by weight of an alkyl acrylate, alkyl methacrylate, vinyl acetate, styrene, acrylonitrile or mixtures thereof; and
- up to 10 parts by weight of a flame retardant synergist per hundred parts of fiber; drying the product at a temperature in the range of from about 140° to about 200°F. and then curing by heating at a temperature in the range of from about 250° to about 310°F. for from about 1 to about 5 minutes.
13. The method of claim 12 which comprises blending
- 65 to 95 percent by weight of polyester fiber and
 - 5 to 35 percent by weight of fibers of syndiotactic polyvinyl chloride and then treating the blend with from 5 to 50 percent by weight, based on the weight of fibers of a copolymer of
- from about 60 to 90 percent by weight of vinylidene chloride;
 - from about $\frac{1}{2}$ to about 2.5 percent by weight of N-methylolacrylamide, N-methylolmethacrylamide, N-methylol-4-pentenoguanamine or mixtures thereof;
 - up to about 2.5 percent by weight of acrylamide, methacrylamide, acrylic acid, methacrylic acid, itaconic acid or mixtures thereof;
 - from about 9.5 to about 40 percent by weight of lower alkyl acrylate, lower alkyl methacrylate, vinyl acetate, styrene or acrylonitrile; and

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up to about 10 parts by weight of a flame retardant synergist containing halogen, antimony, phosphorous, nitrogen or an oxide of antimony, arsenic, aluminum, boron or zinc per hundred parts of fiber; drying the product at a temperature in the range of from about 140° to about 200°F. and then curing by heating at a temperature in the range of from about 250° to about 310°F. for

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from about 1 to about 5 minutes.

14. An article of manufacture which comprises the non-flammable, polyester textile of claim 1.

15. An article of manufacture according to claim 14 which comprises the non-woven product of claim 2.

16. An article of manufacture according to claim 14 which comprises the woven product of claim 3.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

PATENT NO. : 3,870,590

DATED March 11, 1975

INVENTOR(S) :Melvin D. Hirwitz

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, line 46 - Delete "of" and in lieu thereof add
-- or --.

Column 7, line 8 - Delete "3-3/4" and in lieu thereof add
-- 3-1/2 --.

Column 7, line 39 - Delete "SbO₅" and in lieu thereof add
-- Sb₂O₅ --.

Column 7, line 45 - Delete "SbO₅" and in lieu thereof add
-- Sb₂O₅ --.

Signed and sealed this 20th day of May 1975.

(SEAL)

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents
and Trademarks